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DISLOCATIONS AND THE TENSILE STRENGTH OF POLYCRYSTALLINE MAGNESIUM OXIDE

Eighteenth Technical Report to O. N. R.

by

R. J. Stokes and C. H. Li

Office of Naval Research Project
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January 1963

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TABLE OF CONTENTS

ABSTRACT

The tensile strength of polycrystalline magnesia can be high (approximately 30, 000 psi) providing there are no 'fresh' or mobile dislocations present. These dislocations may originate in one of two ways. In the first, they may be introduced directly by mechanical contact with the surface. In the second, they may be generated indirectly through the stress concentrations associated with pores. To attain tensile strengths higher than 10-15, 000 psi it is necessary to use fully dense pore free material whose surface has been chemically polished or otherwise protected from mechanical contact.

I. INTRODUCTION

In previous reports[1] [2] it was shown that surface condition was extremely important in determining the tensile strength of single crystals and bi-crystals of magnesium oxide. This work suggested that a similar procedure with comparable attention to detail should be used to evaluate the potential intrinsic tensile strength of polycrystalline magnesium oxide. In this report we present preliminary data on the effect of surface condition and heat treatment on the tensile strength of high density polycrystalline magnesia. It must be re-emphasized that the term surface condition in the present context does not mean the presence or absence of 'microscopic' damage of the kind introduced during cutting and grinding operations which takes the form of cleavage or intergranular cracking in the surface grains but rather submicroscopic damage of the kind described in the previous papers [1] [2] which causes a change in the type of dislocation (i. e., whether 'fresh' and mobile or not) available to initiate slip. Consequences of 'microscopic' surface damage are already well known and obviously result in a deterioration in strength as described elsewhere[3, 4, 5, 6]. The consequences of submicroscopic surface effects have not previously been considered.

II. EXPERIMENTAL PROCEDURE

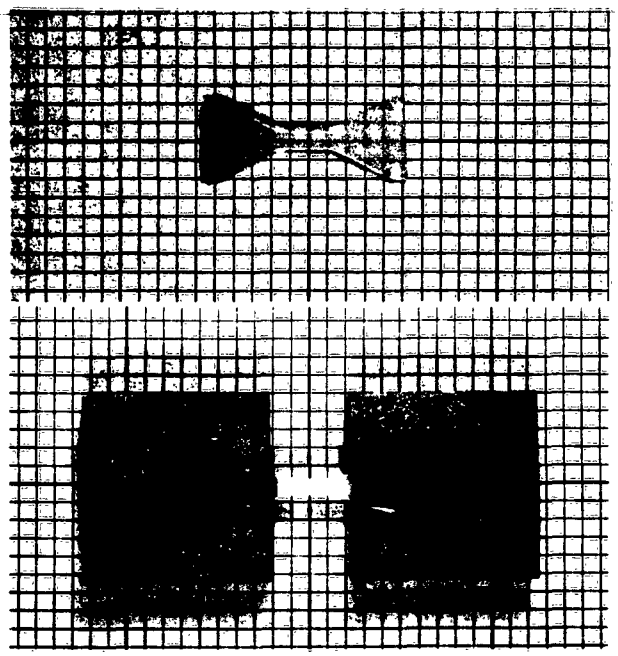
Two sources of material have been investigated, one hot pressed and the other sintered high density magnesia.

The high density hot pressed samples (designated H1 and H2) were kindly provided by Dr. R. M. Spriggs of Avco Corporation, Wilmington, Massachusetts. Both were hot pressed in air in conventional graphite dies to densities of 3.581 or 100 percent of theoretical. This material was fairly transparent initially, as can be seen in Figure 1(a). The samples were sliced with a diamond saw and then cut ultrasonically to the shape shown in Figure 1(a), with the tensile axis perpendicular to the pressing direction and the gauge section taken from the center of the pressing where the density was highest.

High density sintered samples (designated S1 and S2) were kindly supplied by W. B. Harrison of the Honeywell Research Center. They were prepared by an isostatic pressing and sintering technique[6] and had densities which varied from 98 to 99.1 percent depending upon the processing. More recently, material of higher density has been obtained but it was not available at the time of these tensile tests. They were provided in the form of one and a half inch square by 1/4" thick tiles which were also sliced with a diamond saw and cut ultrasonically to the profile of Figure 1(a).

Some of the samples from both sources were annealed at 2000° C in a carbon resistance furnace before the cutting operations.

All of the high density polycrystalline specimens were chemically polished in boiling 85 percent orthophosphoric acid for a period of 15 minutes. They were then washed in boiling distilled water, rinsed in cold distilled water, alcohol and ether and dried to leave a highly polished surface free from any chemical deposit. During the chemical polishing they were suspended by platinum wires from their ends and great care was taken not to touch the reduced gauge section.



(a)

(b)

Figure 1 - Tensile specimen of hot pressed polycrystalline magnesium oxide

(a) unmounted

(b) mounted

The specimens were mounted in the tapered tensile grips illustrated in Figure 1(b) with epoxy cement and loaded in uniaxial tension in the Instron machine with the cross head deflection rate set at .002" per minute.

After the tensile tests a study was made of the characteristic fracture surfaces. Direct examination was often complicated by the extreme roughness and uneven reflectivity of the various ceramic grains. To overcome this, collodian replicas were prepared of the fracture surfaces[7]. The collodian replicas were coated with chromium and then, because the replica surfaces were still not flat enough to be examined easily in the microscope, the replicas were placed face down on a microscope slide and pressed flat while their backs were in contact with adhesive tape. This procedure gave slightly distorted replicas of the fracture surfaces but large areas were within reasonable focus and very convenient for examination and photomicrography. The distortion was not serious enough to be detectable at 500X.

III. MICROSTRUCTURE

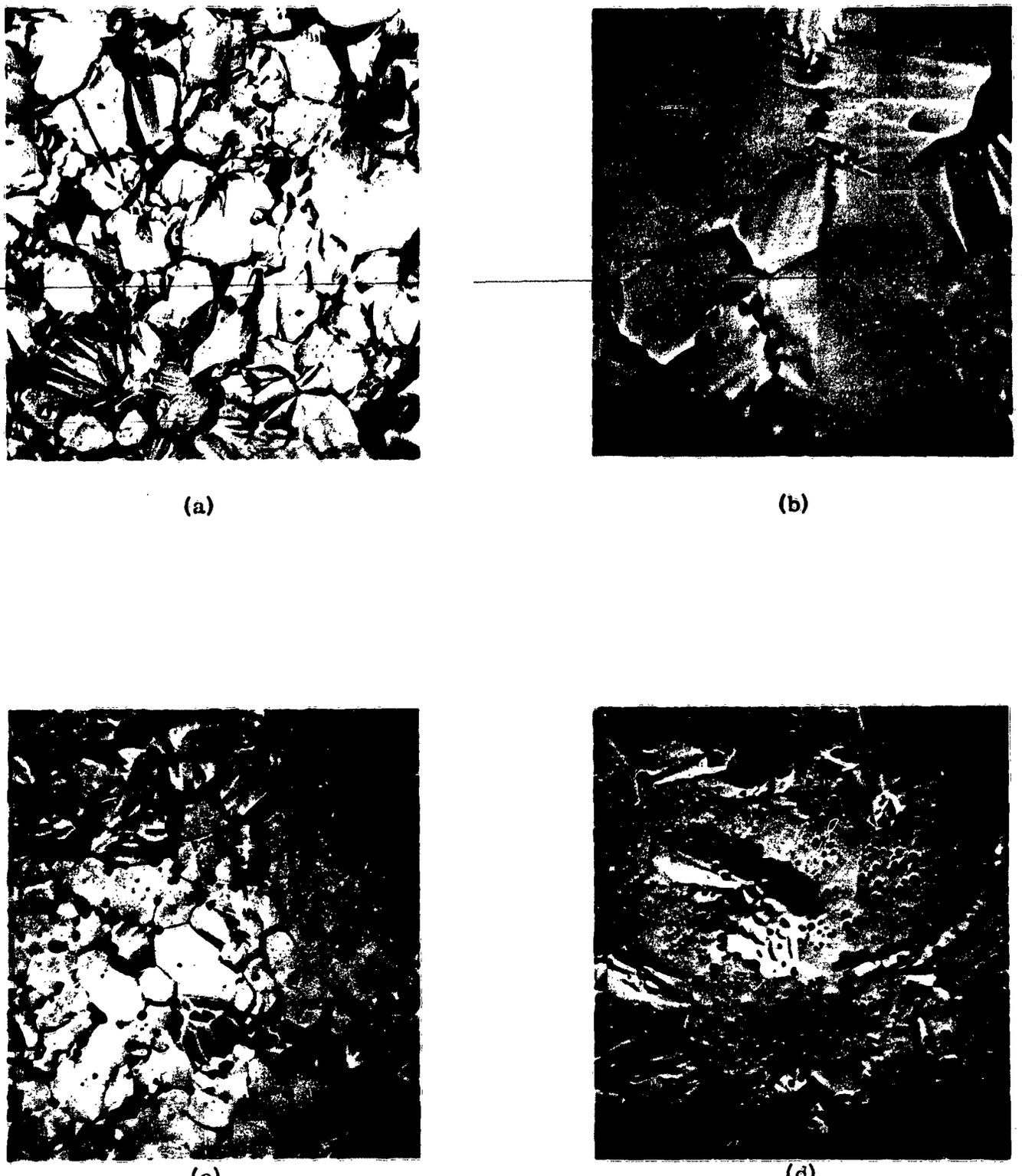
A. HOT PRESSED MATERIAL

The two samples of hot pressed magnesia (designated H1 and H2) both had the same initial microstructure. They will be considered together. The surface appearance after chemical polishing is illustrated in Figure 2(a). The roughness was due to the different rates of chemical attack for grains of different orientation and the higher rate of attack at grain boundaries. The initial grain size was 10-30μ and no porosity could be resolved either within the grains or at the intergranular interfaces under the microscope. There was however a second phase present along the triple lines where grains intersected. This phase was not soluble in orthophosphoric acid and consequently could be found after the chemical polish as a fine skeleton of the triple lines exposed above the surface, pieces of this skeleton which have collapsed on the polished surface are indicated at X in Figure 2(a).

The second phase disappeared during the anneal at 2000° C probably by diffusion along the triple lines and evaporation at the surface. This resulted in some porosity in the intergranular surfaces. The pores were responsible for the open pits in the polished surface of Figure 2(b). The grain size increased during the anneal to 75μ.

B. SINTERED MATERIAL

The two samples of sintered magnesia (designated S1 and S2) had microstructures very different from the hot pressed material. The microstructure of sample S2 is reproduced in Figure 2(c). It had a density of 99. 1 percent and the grain size was between 10 and 20μ. Most of the porosity was located along triple lines with some in the integranular surfaces and some entrapped within the grains. Sample S1 had a density of 98 percent and a mean grain diameter of 5μ. The distribution of the porosity was basically the same as S2, except that a greater proportion was located in the intergranular surfaces and within the grains.



(a) (b)

(c) (d)

Figure 2 - Surface appearance of polycrystalline magnesium oxide specimens after chemical polishing (all x 500). (a) Hot pressed, as-received, Specimen H1-8; (b) Hot pressed, annealed 2000° C, 1/2 hour, Specimen H2-6; (c) Sintered as-received, Specimen S2-3; (d) Sintered, annealed 2000° C, 1/2 hour, Specimen S1-5.

Annealing either sample S1 or S2 at 2000° C for 1/2 hour resulted in a four-fold increase in their respective grain sizes with most of the porosity becoming entrapped within the grains. The result of annealing the very fine grained sample S1 is illustrated in Figure 2(d).

Figure 2 essentially contains examples of the four types of microstructure investigated briefly in this work. Figure 2(a) is an example of fully dense, pore free material, (b) is an example of high density material with the pores located exclusively at the grain boundaries, (c) with the pores located primarily along triple lines and within some grains, (d) with the pores located almost exclusively within the grains.

IV. TENSILE TESTS

The tensile strengths of hot pressed and sintered magnesia specimens are included in Tables I and II respectively together with another pertinent data.

A. EFFECT OF SURFACE CONDITION ON THE TENSILE STRENGTH OF HOT PRESSED MAGNESIA

In this section we are concerned with comparing the results in Tables I(a) and I(b). Both of these groups of specimens were tested in the as-received condition and therefore had the microstructure represented in Figure 2(a). The only difference between them was that specimens of Table I(b) were deliberately sprinkled with fine 200 mesh silicon carbide powder before testing, a procedure known to introduce 'fresh' dislocation sources into the surface of single crystals.[2] The sprinkling did not result in any recognizable deterioration in the surface appearance, in fact Figure 2(a) was taken on a specimen which had been sprinkled.

The outstanding feature of the results was first the extremely high strength of as-received material and second the marked lowering of the strength following the sprinkling treatment. The fracture strength averaged over eight chemically polished as-received specimens was 28, 140 psi with the strongest specimen fracturing at a tensile stress of 35, 255 psi (Spec. H2-3). Assuming the modulus of rupture to be approximately double the uniaxial tensile strength[8] then the average fracture strength corresponded to a modulus of rupture value of 56, 000 psi or approximately four times the value normally quoted for magnesium oxide[9]. It also corresponded to a stress level approximately four times the stress to move fresh dislocations in single crystals of this material.[2][10]

After sprinkling, the tensile strength dropped by approximately one third to an average value of 18, 690 psi as shown in Table I(b). It was tempting to conclude that the drop in strength was a direct consequence of the introduction of 'fresh' dislocations as was shown to be the case for single crystals and bi-crystals in the previous papers[1][2]. Attempts were made to obtain direct evidence to support

TABLE I

EFFECT OF HEAT TREATMENT AND SURFACE CONDITION ON THE TENSILE STRENGTH OF HOT PRESSED MAGNESIUM OXIDE

Sample and Specimen No.	Density (%)	Grain Size (μ)	Microstructure	Fracture Strength (psi)	
		(a) As Received - Chemically Polished			
H1-1	100	5-30	No pores. Second phase along	21, 725	
H1-2	"	"	triple lines.	25, 200	
H1-3	"	"		26, 660	
H1-4	"	"	"	30, 050	
H1-5	"	"		34, 200	Average 28, 140 psi
H2-1	100	5-30	No pores. Second phase along	21, 750	
H2-2	"	"	triple lines.	30, 300	
H2-3	"	"	"	35, 255	
		(b) As Received - Chemically Polished and Sprinkled			
H1-6	100	5-30	No pores. Second Phase along	13, 200	
H1-7	"	"	triple lines	16, 000	
H1-8	"	"	"	16, 500	Average
H1-9	"	"		19, 750	18, 690 psi
H1-10	"	"	"	22, 350	
H1-11	"	"		24, 340	
		(c) Annealed 2000° C 1/2 hour - Chemically Polished			
H1-12	100	75	Slight porosity along triple	16, 200	
H1-13	"	"	lines. None within grains.	18, 800	
H1-14	"	"		22, 000	Average
H2-4	"	"	"	16, 850	19, 410 psi
H2-5	"	"		18, 560	
H2-6	"	"	"	24, 050	
		(d) Annealed 2000° C 1/2 hour - Chemically Polished and Sprinkled			
H1-15	100	75	Slight porosity along triple	10, 040	
H1-16	"	"	lines. None within grains.	12, 800	
H1-17	"	"		15, 150	Average
H1-18	"	"	"	15, 700	14, 930 psi
H1-19	"	"		20, 950	

NOTE: Tensile Stress for plastic deformation of single crystals is 10, 000-12, 000 psi

this interpretation using the etch pit technique for revealing slip. However, careful examination of all surface grains suitably oriented for etching yielded no conclusive evidence that slip had occurred prior to fracture in any of them. A side experiment, in which the polycrystalline specimen surface was deliberately scratched and then etched, confirmed that the etch was capable of revealing slip dislocations in polycrystalline material when they were present.

While direct experimental evidence is lacking at the moment, it is nevertheless believed that the reduction in strength after sprinkling is due to the introduction of 'fresh' mobile dislocations. These result in limited slip in a few grains and the nucleation of fracture by dislocation pile up at grain boundaries in the manner shown for bi-crystals[1][11][12][13]. The primary reason for this belief is the reasonable agreement between the fracture strength of sprinkled polycrystalline material (18, 500 psi) and the yield strength of single crystals (10, 000 psi) and the fracture strength of sprinkled bi-crystals (10, 000-20, 000 psi)[1][2].

Whatever the fundamental reason for the reduction in strength, these experiments clearly demonstrate the extreme sensitivity of high density polycrystalline magnesium oxide to surface condition. Whereas in the past the effect of surface damage on the strength of ceramics has always been regarded as a consequence of surface flaws acting as Griffith cracks which propagate elastically at a certain critical stress, there is now a need to advance a step further and consider the effect of dislocations introduced into the surface layers of fully dense material which generate slip bands to interact with grain boundaries and nucleate fracture. It is disturbing to realize that if the potentially high strength of fully dense hot pressed magnesium oxide is to be exploited then careful consideration must be given to procedures for preparing and protecting surfaces to high degrees of perfection.

B. EFFECT OF HEAT TREATMENT ON TENSILE STRENGTH OF HOT PRESSED MATERIAL

The tensile strength of hot pressed magnesium oxide was also compared before and after an anneal at 2000° C for 1/2 hour. The anneal resulted in a change in microstructure from that illustrated in Figure 2(a) to Figure 2(b) and a drop in strength by about one third from an average value of 28,140 psi to 19,410 psi. This can be seen by comparing Tables I(a) and I(c).

The reason for this deterioration must be associated with the change in microstructure since the surface condition is the same. It is interesting to consider which of the two parameters, grain size or porosity, are more likely to be responsible for the loss in strength from the point of view of dislocation theory. First, we consider grain size. The grain size effect observed in brittle metals is generally interpreted[14] in terms of the number of slip dislocations which are able to emanate from a given slip source before the back stress due to dislocation pile up at a grain boundary causes it to cease. The larger the grain diameter the greater the number of dislocations per slip source which can be generated to pile up under a given applied stress. This results in a more severe local tensile stress concentration and eventually in a lower fracture strength. It is implicit in this interpretation of the grain size effect that 'fresh' or mobile dislocation sources are present and able to multiply, the mere change in grain size by itself does not result in any marked change in fracture strength. This is important because in many ceramic materials the dislocations are not mobile at room temperature and direct application of the theories developed for brittle metals is not justified. In particular in the present case for magnesium oxide, an anneal at 2000° C has been shown in single crystals[1][2] to eliminate mobile dislocation sources. For this reason the increase in grain size from that of Figure 2(a) to Figure 2(b) is not considered to be the most important factor responsible for the drop in strength.

Instead, it is considered that pores produced by the heat treatment play the most important role. It is proposed that the stress concentrations associated with the pores assist the applied stress in nucleating 'fresh' dislocations either within the grains or at intergranular interfaces. These mobile dislocations multiply to generate a slip band and nucleate a crack by dislocation pile up in the manner shown in the experiments on bi-crystals.[1][11][12][13] The fact that the drop in strength upon annealing is approximately equal to the drop in strength following sprinkling (compare Tables I(b) and I(c)) tends to substantiate this interpretation. In other words, in the absence of microscopic surface damage, the strength of high density polycrystalline magnesia is limited by the presence of even a slight amount of porosity. To attain high tensile strengths it is absolutely necessary to utilize pore free material such as the as-received hot pressed material.

If the above proposition is correct then sprinkling an annealed (2000° C) hot pressed specimen should not result in any further marked drop in strength. If 'fresh' dislocations are already available from the pores, introducing them deliberately into surface layers by sprinkling should make no difference. Results of experiments conducted to investigate this point are included in Table I(d). They show that the average strength of sprinkled annealed material (14, 930 psi) is only slightly less than the average strength of unsprinkled annealed material (19, 410 psi).

C. THE TENSILE STRENGTH OF SINTERED MATERIAL

The tensile strengths of sintered magnesia specimens are included in Table II. Both samples had approximately the same average strength, 15, 500 psi for S1 and 14, 700 psi for S2. Again, assuming the modulus of rupture to be double the uniaxial tensile strength, the modulus of rupture should be approximately 30, 000 psi in agreement with independent measurements on similar samples under transverse bending[6].

TABLE II

THE TENSILE STRENGTH OF SINTERED MAGNESIUM OXIDE

Sample and Specimen No.	Density (%)	Grain Size (μ)	Microstructure	Fracture Strength (psi)	
		(a) As-Received - Chemically Polished			
S1-1	98	1-5	Pores in grain boundary interfaces and pores within grains.	12, 700	Average 15, 470 psi
S1-2	"	"		15, 300	
S1-3	"	"		16, 500	
S1-4	"	"	"	17, 375	
		(b) Annealed 2000° C - Chemically Polished			
S1-5	98	20-50	Pores within grains.	12, 375	
		(c) As-Received - Chemically Polished			
S2-1	99. 1	10-20	Pores along triple lines.	10, 150	Average 14, 690 psi
S2-2	"	"	Slight porosity within grains.	16, 075	
S2-3	"	"	"	17, 850	
		(d) Annealed 2000° C - Chemically Polished			
S2-4	99. 1	100	Pores within grains	11, 100	

NOTE: Tensile Stress for plastic deformation of single crystals is 10, 000-12, 000 psi.

After annealing at 2000° C the microstructure changed from that represented in Figure 2(c) to 2(d). Both samples showed a slight decrease in strength. Unfortunately, there were insufficient specimens to pursue the effect of heat treatment any further.

The low tensile strength of as-received sintered material compared with as-received hot pressed material (compare Tables I(a) and II) was presumably due to the presence of porosity. The fact that its strength was generally lower than annealed hot pressed material (compare Tables I(c) and II) had no immediate explanation. Apart from a greater amount of porosity and smaller grain size the chief microstructural difference was in the location of the pores. In the sintered material some pores were located within the grains and could have been more effective in nucleating slip there.

Since sintered material almost inevitability contains some porosity it seems unlikely that it will be possible to achieve the same high tensile strengths which can be reached with hot pressed material.

It was interesting to note that the specimens containing mobile disolcations, either because they were generated at pores or introduced deliberately at the surface, showed very little variation in strength with grain size. Thus the tensile strength of sprinkled single crystals and bi-crystals (approximately 10,000 psi.[1]) annealed hot pressed material (Table I(c)) and fine grained sintered material (Table II) all varied between 10,000 psi and 18,000 psi and this variation showed no simple dependence on either grain size or porosity. This lends support to the conclusion expressed elsewhere[15] that the dislocation density in a slip band in magnesium oxide at room temperature is so high that any improvement in strength due to grain size refinement will not become apparent until the grain size is decreased well below 10μ. As stated before the best avenue for a marked improvement in strength is through the immobilization of dislocations.

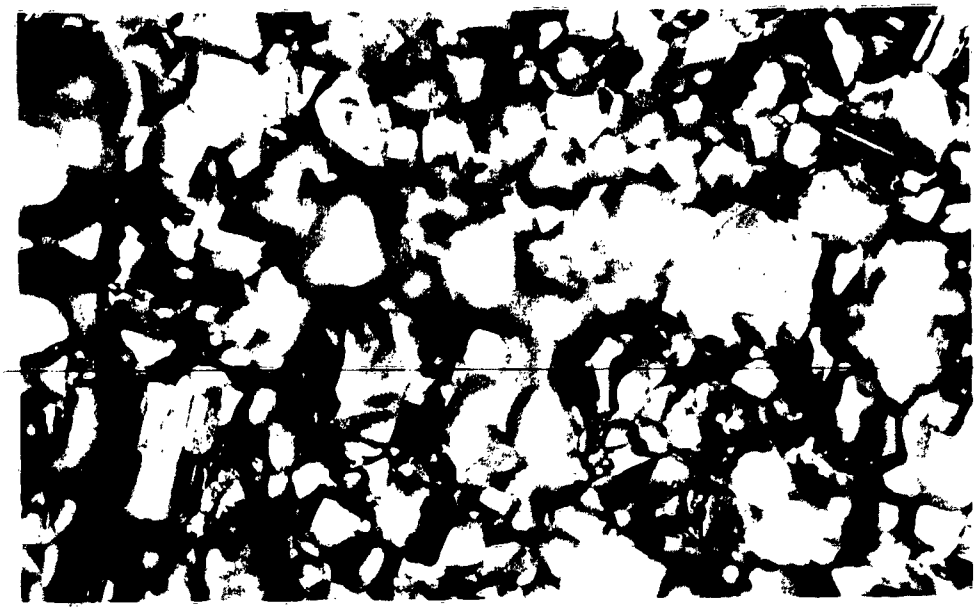
V. FRACTURE SURFACE STUDIES

The fracture surfaces of all the specimens in Table I and II were examined either directly or indirectly through the use of the collodian replica technique described earlier.

Figures 3(a) and 3(b) compare the fracture surfaces of hot pressed material before and after the anneal at 2000° C. In both cases the fracture was predominantly integranular (approximately 90 percent) with the remainder clevage or transgranular fracture. The fracture surface of the as-received hot pressed material in Figure 2(a) was practically featureless, it consisted of smooth curved intergrannular surfaces with the occasional cleavage facet distinguished by its flat surface and straight and often parallel (to $[\bar{1}00]$) cleavage markings. There was no evidence of porosity in agreement with the microstructure of Figure 2(a). In Figure 3(b) the curved intergranular surfaces were no longer smooth but pitted from the pores originally present. No pores were observed on the cleavage facets confirming that the porosity was restricted to the intergranular interfaces.

The fracture surfaces of the sintered specimens similarly reflected their microstructure. The proportion of intergranular to cleavage (90:10) was about the same as for hot pressed material. Figure 3(c) reproduces the fracture surface of the sintered sample S1 before annealing and Figure 3(d) after annealing. Figure 3(d) should be compared with the microstructure of Figure 2(d). In both of these specimens pores were visible on the cleavage facets as well as the intergranular surfaces.

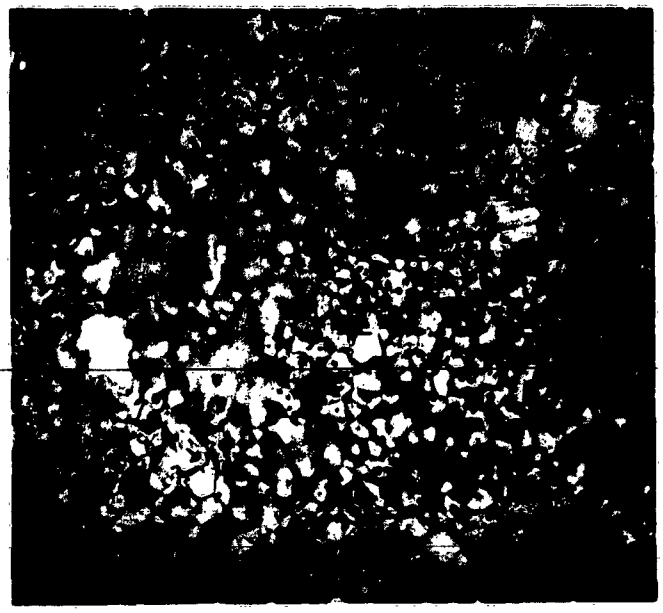
Because of the lack of tear markings on the fracture surfaces it was not possible to identify the fracture source. If fracture had occurred entirely by cleavage then the cleavage tear lines could have been traced back to their origin, but with the relatively featureless integranular surfaces it was not possible to do this. However, on the basis of the behavior of other fully dense ionic solids of the same lattice structure[15] and the observations on bi-crystals described earlier[1] it was reasonable to assume that the origin of fracture was an intergranular rupture. Fracture then followed the path of least resistance which according to Figure 3 was predominatly intergranular for the material tested in the present work.



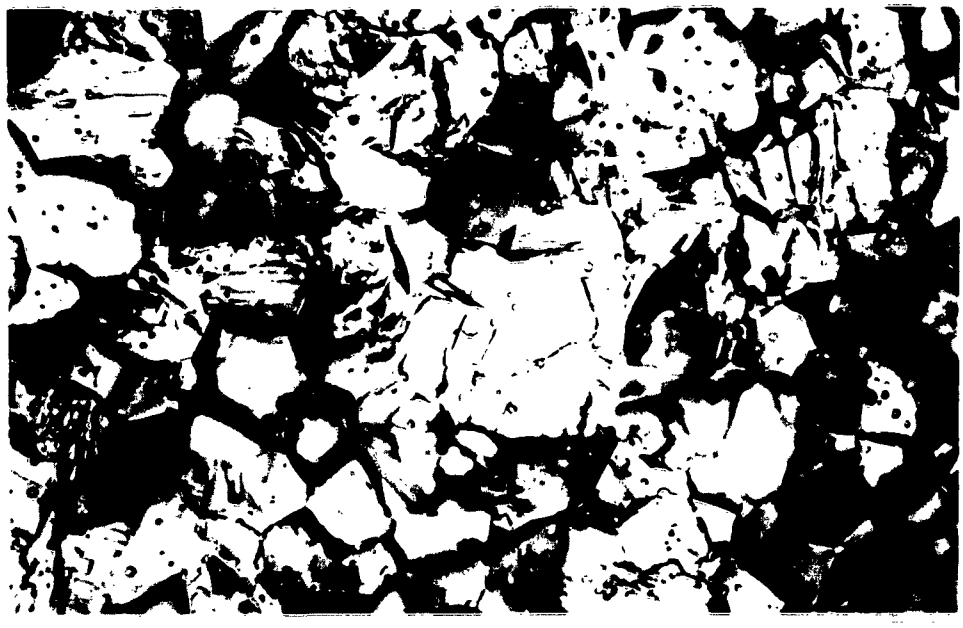
(a) Hot pressed, as-received - Specimen H2-2 (X500)

(b) Hot pressed, annealed 2000° C, 1/2 hour, Specimen H2-6 (X500)

Figure 3 - Photomicrographs taken on replicas of polycrystalline magnesium oxide fracture surfaces.



(c) Sintered, as-received, Specimen S-1 (X625)



(d) Sintered, annealed 2000° C, 1/2 hour, Specimen S1-5 (X500)

Figure 3 - Photomicrographs taken on replicas of polycrystalline magnesium oxide fracture surfaces.

VI. DISCUSSION

The main points of this paper and the previous papers on single and bi-crystals[1][2] may be summarized as follows:

(i) Research on single crystals has shown that the mechanical properties of magnesium oxide falls into two categories, the choice of which depends on the availability of mobile dislocations. Single crystals are either extremely strong and elastic in the complete absence of mobile dislocation sources or relatively weak and ductile in their presence. It has been found that these mobile dislocation sources are 'fresh' dislocation loops injected into the crystal by mechanical contact with the surface[2].

(ii) The mechanical behavior of bi-crystals also falls into two categories. In the absence of dislocation sources bi-crystals are extremely strong and elastic like the single crystals but in the presence of mobile dislocations they are relatively weak and brittle. The brittleness is due to the direct interaction of slip bands with grain boundaries to generate cracks[1]. The only way in which this brittleness may be avoided is for the grain boundary to have a small misorientation[13].

(iii) The strength of polycrystalline magnesium oxide is also sensitive to the presence of mobile dislocation sources. When care is taken to eliminate surface defects and to immobilize dislocations, tensile strengths of 30, 000 psi can be attained, but when mobile dislocations are present the strength drops to a value between 15, 000 psi and 20, 000 psi. These mobile dislocations can be introduced directly by mechanical contact with the surface or indirectly through the presence of pores.

With these points in mind there arises the question of what steps might be taken to enhance the mechanical properties of polycrystalline magnesia. If ductility is to be achieved then obviously polycrystalline material must be developed with a highly preferred texture such that the misorientation from grain to grain is small and the tendency for crack formation reduced. The cubic structure of magnesium oxide would be advantageous in this case since the mechanical properties would still be reasonably isotropic.

The other approach is to avoid plasticity altogether and try to attain the extremely high tensile strengths which have been measured on single crystals (160, 000 psi)[2] and bi-crystals (110, 000 psi)[1]. To do this, procedures will have to be developed either to eliminate dislocation sources or to make dislocations less mobile. The elimination of dislocation sources calls for material free of pores on the one hand and protection of the surface on the other. Now that fully dense material has become available protection of the surface has become a factor of paramount importance. The use of surface coatings or diffused surface layers resistant to dislocation motion should be considered.

The mobility of a dislocation depends on a number of factors such as crystal structure, bond character, temperature and microstructure. Unfortunately, the crystal structure, etc., of pure magnesium oxide at room temperature is such that dislocations are very mobile at comparatively low stresses[10]. In materials such as alumina which have a more complex crystal structure and correspondingly complex dislocation configuration[16] the stress to move dislocations at room temperature is exceedingly high. Because of this one would expect surface condition to be far less important in determining the strength of high density alumina. To change the mobility of dislocations in magnesia demands a change in microstructure or bond character possibly by alloying. The mechanical behavior of magnesium oxide alloys as a function of heat treatment is currently under study in our laboratory with a view to learning how to lock dislocations or increase the lattice resistance to their motion, these results will be published later.

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